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Indian Standard

PESTICIDE RESIDUES IN FOODS — CYPERMETHRIN, DELTAMETHRIN, FENVALERATE AND PERMETHRIN — METHOD FOR DETERMINATION

भारतीय मानक

खाद्य पदार्थों में कीटनाशी अवशिष्ट — साइपरमेथ्रीन, डेल्टामेथ्रीन, फेंवैलरेट एवं परमेथ्रीन – ज्ञात करने की पद्धति

UDC 664 : 543 : 632·951·028 CYP

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1989

Price Group 2

Pesticides Residue Analysis Sectional Committee, AFDC 56

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 23 February 1989, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Agricultural and Food Products Division Council.

Synthetic pyrethroids, such as, cypermethrin, deltamethrin, fenvalerate and permethrin are used as their respective formulations in agriculture for the control of insect pests. This standard will enable the health authorities and others engaged in these field to follow uniform test procedure for estimation of the residues of the synthetic pyrethroids in various food commodities.

In the preparation of this standard, due consideration has been given to the limits of cypermethrin, deltamethrin and fenvalerate residues laid down under the provisions of *Prevention of Food Adulteration Rules, 1955* and the test method is sensitive to the prescribed level of residues.

In reporting the result of a test or analysis made in accordance with the standard, if the final value, observed or calculated, is to be rounded off, it should be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

PESTICIDE RESIDUES IN FOODS — CYPERMETHRIN, DELTAMETHRIN, FENVALERATE AND PERMETHRIN — METHOD FOR DETERMINATION

1 SCOPE

1.1 This standard prescribes the gas chromatographic method for determination of synthetic pyrethroids, namely, cypermethrin, deltamethrin, fervalerate and permethrin residues in various food commodities.

1.1.1 This method is applicable with a limit of detection in the range of 0·005 — 0·05 µg/g (0·005 — 0·05 ppm).

1.1.2 Though no set procedure for thin layer chromatography (TLC) is being prescribed, standardized TLC may be followed, if necessary, for the purpose of identification and confirmation of residues.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 1070 : 1977	Specification for water for general laboratory use (*second revision*)
IS 11380 : 1985	Methods of sampling for the determination of pesticide residues in agricultural and food commodities

3 QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4 SAMPLING

4.1 The representative samples for the purpose of estimating cypermethrin, deltamethrin, fenvalerate and permethrin residues in food commodities shall be drawn in accordance with IS 11380 : 1985.

5 APPARATUS

5.1 Deep-Freezer

5.2 Waring blender

5.3 Refrigerator

5.4 Air-Oven

5.5 Rotary Vacuum Evaporator

5.6 Chromatographic Column, glass, 400 mm length, 15 mm ID, with 100-ml reservoir and stop-cock arrangement.

5.7 End-Over-End Shaker or Horizontal Soil Shaking Machine

5.8 Buchner Funnel

5.9 Gas Chromatograph

Any suitable model equipped with electron capture detector (Ni 63 ECD). The operating parameters, as suggested below, may be varied, according to the available facilities provided standardization is done:

Column : Glass, 1000 mm long, 2 mm ID, packed with 1·5 percent SP 2250 and 1·05 percent SP 2401 on suplecoport

Temperature
Column : 230°C
Detector : 270°C
Carrier gas : Nitrogen

Carrier Gas Flow Rate : 50 ml/minute

NOTE — The order of elution will be permethrin, cypermethrin, fenvalerate and deltamethrin.

6 REAGENTS

6.1 Acetonitrile, all glass redistilled.

6.2 *n*-Hexane, all glass redistilled.

6.3 Diethyl Ether, all glass redistilled.

6.4 Ethyl Acetate, all glass redistilled.

6.5 Sodium Sulphate, anhydrous, AR grade.

6.6 Silica Gel G, 60-120 mesh, AR grade, activated at 110°C for 6 hours.

6.7 Cypermethrin Reference Standard, of known purity.

6.8 Deltamethrin Reference Standard, of known purity.

6.9 Fenvalerate Reference Standard, of known purity.

6.10 Permethrin Reference Standard, of known purity.

7 SAMPLE PREPARATION

7.1 Cereals, Pulses, Condiments, Tea, Coffee, Sugar, etc

If needed, grind to pass through 750-micron IS sieve. Reduce to about 100 g by mixing and quartering.

7.2 Vegetables and Fruits

7.2.1 Remove all visible soil particles by gentle rubbing. Scrub root vegetables with a brush and wash.

7.2.2 Remove all inedible portions of vegetables, such as, outer leaves of cabbages, carrot tops, stems of tomatoes, stones of fruits, etc. Remove the peel of the skin of a fruit if it is normally not consumed, such as, peas, oranges, etc. Do not remove the skin if it is edible, such as, apples, carrots, etc.

7.2.3 Cut a representative sample into pieces and reduce to about 100 g by mixing and quartering.

7.3 Oilseeds and Nuts

Remove and discard shells, if present. Reduce the sample to the desired size for extraction by mixing and quartering. Chop the sample finely.

7.4 Hydrogenated and Unhydrogenated Vegetable Oils

These do not require any preparation.

7.5 Water and Beverages

These do not require any preparation.

8 SAMPLE STORAGE

Store samples as such or if the bulk is too much, after the preparation of the sample. Depending upon the nature of the sample, keep the samples or their extracts either in deep freezer at — 15°C or in refrigerator until taken up for analysis. Ensure that the samples do not absorb or lose moisture during storage. Avoid undue long storage periods.

9 EXTRACTION

9.1 Non-Fatty Foods Containing Less Than 2 Percent Fat

9.1.1 Extract 50 g of the finely chopped sample twice with 150 and 100 ml (65 : 35) acetonitrile-water mixture and 50 g sodium sulphate, by blending for 2 minutes each time.

9.1.2 Either extract by the procedure described in **9.1.1** or macerate the sample in a blender and extract with 150 ml and 100 ml of acetonitrile by shaking for 30 minutes, each time, in an end-over-end shaker or horizontal soil shaking machine. Filter the extracts through a Buchner funnel using a Whatman No. 1 or equivalent filter paper under suction. Rinse twice with 25 ml of acetonitrile-water (65 : 35) mixture and collect the filtrate. Concentrate the extract to about 150 ml in a rotary vacuum evaporator.

9.2 Fatty Foods Containing More Than 2 Percent Fat

9.2.1 *Oils*

Dissolve 10 g of the sample of 100 ml hexane and partition twice into 125 ml of hexane saturated acetonitrile, each time collecting the lower layer. Concentrate the combined acetonitrile layer to about 50 ml.

9.2.2 *Oilseeds, Nuts, Etc*

Blend 25 g sample for 5 minutes at high speed with 250 ml acetonitrile-water (85 : 15) mixture. Allow the suspension to settle and filter under vacuum collecting the filtrate in a flask. Place the filtrate in a deep-freezer at −15°C until the water and fats are frozen and then decant the supernatant acetonitrile phase (equivalent to 10 g of sample aliquot).

9.3 Water, Beverages, Etc

Partition 1 litre of the sample into 100, 100 and 50 ml hexane-ether (8 : 2) mixture and dry the combined extracts through sodium sulphate and concentrate in a rotary vacuum evaporator over a water-bath maintained at 50°C.

10 LIQUID-LIQUID PARTITIONING

10.1 Non-Fatty Foods Except for Water and Beverages, Etc

Take the extract in a 1-litre separatory funnel. Dilute with 450 ml of 5 percent aqueous sodium chloride solution and extract with 200, 100 and 50 ml portions of hexane-ether (8 : 2) mixture shaking the separatory funnel vigorously for 2 minutes and filter the combined extract through sodium sulphate. Rinse the sodium sulphate layer twice, each time with 25 ml of hexane-ether mixture. Evaporate the combined extract to near dryness on a rotary vacuum evaporator at 35°C and dissolve the extract in 5 ml hexane.

10.2 Fatty Foods

Transfer the filtered sample-extract-aliquot to a 1-litre separatory funnel, add 100 ml hexane and shake for 30 seconds. Add 400 ml of 2 percent aqueous sodium chloride solution and shake again for one minute. Allow the phases to separate. Rinse the hexane phase with 50 ml water. Dry the hexane phase through 50 g sodium sulphate, concentrate below 50°C in a rotary vacuum evaporator and dissolve the extract in 5 ml hexane.

11 CLEAN UP

Wet pack 5 g sodium sulphate, 25 g silica gel and 10 g sodium sulphate, bottom upward, in the glass column using hexane and pre-wash with 50 ml hexane. Add the concentrate (*see* **9.3, 10.1** or **10.2**) to the column and just as the extract drains into the sodium sulphate layer, elute with 200 ml of hexane-ethyl acetate (95 : 5) mixture. Evaporate the elute below 50°C in a rotary vacuum evaporator and take a suitable volume for gas chromatographic estimation.

12 GAS CHROMATOGRAPHY

12.1 Prepare working standard solutions of each of the reference standards (*see* **6.7** to **6.10**) to give concentrations of 0·1, 0·2, 0·5, 1·0 and 2·0 μg/ml. Suitably adjust the instrument (*see* **5.9**) so as to get at least 50 percent fullscale deflection with a 1 μg injection.

12.2 Prepare a suitable calibration graph of peak area against quantity injected and quantify the unknown by plotting the peak area values. The calibration range is to be selected in such a manner as to ensure that the unknown sample values fall in the mid-range of the calibration curve.

13 CALCULATION

Quantity of cypermethrin/deltamethrin/fenvalerate/permethrin can be estimated as state below:

$$X = \text{residue in } \mu\text{g/g (ppm)} = \frac{A_1 \times M_1 \times V_2}{A_2 \times M_2 \times V_1} \times \frac{100}{R}$$

where

X = Cypermethrin/deltamethrin/fenvalerate permethrin under estimation;

A_1 = peak area of the sample injected;

A_2 = peak area of the standard injected;

M_1 = mass in μg, of the standard injected;

V_2 = volume, in ml, of the sample extract;

V_1 = volume, in μl, of the sample injected;

M_2 = mass, in g, of the sample taken for analysis; and

R = Percent purity of the reference standard.

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